Adhesion behavior of conventional and high-translucent zirconia: Effect of surface conditioning methods and aging using an experimental methodology

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Abstract: Objective: Evaluate the adhesive behavior of conventional and high-translucent zirconia after surface conditioning and hydrothermal aging. Materials and methods: Conventional (ZrC) and high-translucent zirconia (ZrT) specimens were divided into six groups: without surface treatment (ZrC and ZrT), air-borne particle abrasion with 50- m Al2 O3 sized particles (ZrC-AO and ZrT-AO), and tribochemical treatment with 30- m silica modified Al2 O3 sized particles (ZrC-T and ZrT-T). Zirconia specimens were treated using an MDP-containing universal adhesive and bonded to two resins blocks with an adhesive luting cement. Microbar specimens with cross-sectioned areas of 1 mm2 were achieved. Half of the microbars were subjected to hydrothermal aging. Bond strength was evaluated by microtensile bond strength test and statistically evaluated by the Weibull analysis. Results: Roughness of the ZrC-AO and ZrT-AO groups were statistically higher. Bond strength analysis revealed higher bond strength for ZrC-AO and ZrC-T groups compared to ZrT-AO and ZrT-T, respectively. Mixed failure was the most frequent for the mechanically treated groups, while no cohesive failures were obtained. Conclusion: Lower values of bond strength were obtained for the mechanically treated high-translucent zirconia groups when compared to their conventional zirconia counterparts. Mechanical surface treatment significantly improved the bond strength to conventional and high-translucent zirconia. Clinical significance: Mechanical surface treatment (air-borne particle abrasion or tribochemical treatment) associated with the use of universal adhesives containing MDP could provide a durable bonding to conventional and high-translucent zirconia.
Keywords: adhesive cementation; high-translucent zirconia; microtensile bond strength.

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Adhesion to conventional and high-translucent zirconia: effect of different surface conditioning methods and of hydrothermal aging

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Results. Roughness of the ZrC-AO and ZrT-AO groups were statistically higher. Bond strength analysis revealed higher bond strength for ZrC-AO and ZrC-T groups compared to ZrT-AO and ZrT-T respectively. Mixed failure was the most frequent for the mechanically treated groups, while no cohesive failures were obtained.

Conclusion. Lower values of bond strength were obtained for the mechanically treated high-translucent zirconia groups when compared to their conventional zirconia counterparts. Mechanical surface treatment significantly improved the bond strength to conventional and high-translucent zirconia.

Keywords: High-translucent zirconia; Microtensile bond strength; Adhesive cementation.

INTRODUCTION

Due to its superior mechanical properties, yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) is one of the most suitable ceramic materials for use as prosthetic frameworks and implant abutments.[1,2] Although 3Y-TZP (1st generation zirconia) is still the most used by clinicians, the fact that it is white and opaque greatly reduces its clinical indications.[3] In order to minimize such optical limitations, translucent and colored zirconia have been developed by means of microstructural modifications, such as decreased grain size, higher density, increased cubic phase and the addition of coloring oxides.[3–5] Zirconia of the 2nd generation presents a reduction in the alumina concentration and modified sintering parameters, which discretely reduces its degree of opacity.[5] However, an effective gain of translucency and a stable cubic phase is obtained when a higher concentration of yttria (approximately 9.3 wt% / 5mol%) is present, allowing an increment of cubic phase in zirconia composition, decreasing light scattering that occurs at grain boundaries.[3,4] As a result, 3rd generation or high-translucent zirconia developed from this strategy have been used in the manufacture of monolithic crowns and ultrathin restorations, with acceptable aesthetic results.[6,7]

Nevertheless, 3Y-TZP is an acid resistant ceramic due to the absence of silica and the vitreous phase in its composition.[8–10] Thus, to achieve appropriate bonding to zirconia surfaces, both micromechanical and chemical bonding conditioning methods have been suggested, highlighting treatments such as air-borne particle abrasion,[11–14] tribochemical silica coating,[11,15–17] plasma coating with hexamethyldisiloxane,[18] deposition of micro-pearls of low fusing porcelain,[19,20] the use of erbium-doped yttrium aluminium garnet (Er.YAG) laser,[21–23] CO_2 laser, [23] selective infiltration etching (SIE), [24–26] among others.

Air-borne particle abrasion or tribochemical silica coating associated with the application of a hydrophobic phosphoric acid monomer, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), is considered one of the most reliable adhesion bonding protocols for zirconia restorations.[27–31] One-bottle universal adhesives presenting both silane and phosphoric monomers in their composition are indicated to be used in adhesive protocols.[32,33] The MDP

molecules added to these adhesives enable chemical bonding to zirconia through the formation of zirconium phosphate.[34]

Studies have shown predictable outcomes regarding the bond strength of conventional zirconia when mechanical and chemical surface treatments are associated.[9,31,33,35–38] On the other hand, little is known about the adhesive behavior of high-translucent zirconia. Yagawa et al. reported promising results regarding the bond strength in high-translucent zirconia after the use of primers containing MDP; while Salem et al. emphasized the importance of associating mechanical surface treatments and an MDP-containing universal adhesive.[39,40] However, none of these studies compared the adhesive behavior of high-translucent zirconia with the conventional material.

In order to evaluate the bond strength, shear (SBS) and tensile (TBS) tests on macro and micro (μ) scales have been systematically employed.[9,38,41–43] Between these two methods, the SBS test is the preferred method by researchers because of its ease of execution, however, it has been stated that it does not provide a homogeneous distribution of stress at the adhesive interface.[9,38] On the other hand, the μTBS test seems to be the most suitable method to evaluate the bond strength, since it allows a better alignment of the specimens, a better distribution of stress, and a more sensitive analysis of the performance of the cementing agent and its interaction with the material used.[9,38,41,44,45] Nonetheless, the main difficulty of the μTBS test lies in the preparation of the specimens. Due to their reduced size and fragility, loss of specimens during processing is very common, a problem described in the literature as pre-test failure.[46,47] Allied to this, when specimens are made of a more resistant material, such as zirconia, the cutting of the specimens becomes even slower and more difficult, usually leading to a greater loss of specimens, rendering the complete analysis of the data unreachable.[48–50]

To simulate the aging of the bonded interface, thermocycling has been suggested as an effective method.[9,38,51] In addition to the powerful hydrolytic mechanism of water, the thermal cycles allow expansion and contraction of the materials involved, generating fatigue at the interface that could interfere with the adhesion values.[52]

Scientific literature is limited regarding the bond strength of high-translucent zirconia, furthermore, the authors couldn´t find any research

comparing the adhesive behavior between conventional and high-translucent zirconia. Thus, the purpose of the present study was to evaluate the influence of different surface conditioning methods associated to an MDP-containing universal adhesive on the adhesive behavior of conventional and high-translucent zirconia, before and after thermal aging, by means of μTBS testing. An experimental methodology was used to obtain the specimens in order to reduce pre-test failures. The null hypotheses tested were that the bond strength values of high-translucent zirconia after mechanical and chemical treatments would not differ from the values of conventional zirconia; and that thermal aging would not influence the bond strength values for both materials.

MATERIALS AND METHODS

Preparation of the specimens

The study design is presented schematically in Fig. 1. Conventional and high-translucent zirconia ceramic specimens (Upcera; Shenzhen Upcera Dental Technology Co., Liaoning, China) (Table 1) were sintered following the manufacturer's instructions in a furnace (1700 Sinter; EDG Equipamentos e Controles Ltda., São Carlos, Brazil), resulting in final dimensions of 10 x 10 x 1.50 mm after shrinkage. The zirconia specimens were then ultrasonically cleaned (Cristófoli, Paraná, Brazil) in ethanol for 20 minutes to ensure the absence of debris and then air-dried immediately before the surface conditioning method.

Surface conditioning methods

Before adhesive bonding, the surface of conventional and high-translucent zirconia specimens was treated according to Özcan[10] and randomly divided into six groups. The ZrC and ZrT groups did not receive any surface treatment. The surfaces of the ZrC-AO and ZrT-AO groups were air-particle abraded with 50 μm aluminum oxide particles (Bio Art Dental Equipment, São Carlos, Brazil) at 2.5 bar of pressure and at 10 mm distance from the surface for 20 seconds; and in the ZrC-T and ZrT-T groups, tribochemical treatment with silica-coated alumina particles of 30 μm (Rocatec, 3M ESPE; Irvine, CA, USA) was applied to the zirconia surface for 20 seconds at a pressure of 2.5 bar at a distance of 10mm. Surface conditioning methods were performed on both sides of the zirconia

specimens and then they were ultrasonically cleaned in ethanol solution for 20 minutes and air-dried as a previous step to adhesively bond to the resin specimens.

Morphological analysis

Additional conventional and high-translucent zirconia specimens were obtained for all groups (as-sintered, air-particle abraded with 50 µm Al_2O_3 sized particles and tribochemical treatment with 30 µm silica-coated alumina particles) to perform a morphological analysis. The arithmetic average roughness (R_a) of the zirconia surfaces, was evaluated using a stylus profilometer (DektakXT, Bruker Corporation, MA, USA) at six different sites on each specimen. The measurement length was 2 mm and a cut off at 0.25 mm for 3 s.

Zirconia phase fractions of both zirconia materials were quantified using the wide-angle X-ray diffraction (XRD) analysis. XRD spectra were collected using a ray diffractometer (XRD D8 Advance, Bruker Corporation, MA, USA), at 40 kV and 30 mA utilizing CuKα radiation. The range of 2θ angles was from 20 to 80, at a step size of 0.021 and a step time of 2 s.

Bonding and processing procedure

To perform adhesive bonding, resin specimens (Opallis; FGM, Joinville, Brazil) were fabricated with dimensions of 10.0 mm x 10.0 mm x 6.0 mm. These specimens were polished with 400 grit silicon carbide papers and then ultrasonically cleaned in distilled water for 20 minutes. Next, the bonding of the resin to the zirconia specimens was carried out using an MDP-containing universal adhesive (Single Bond Universal, 3M ESPE; Irvine, CA, USA) and an adhesive resin cement (RelyX Ultimate, 3M ESPE; Irvine, CA, USA), according to the manufacturer's instructions. The adhesive cement was placed on both sides of the zirconia specimens and then bonded between two resin specimens under a fixed load of 20N. The excess of resin cement was removed with a brush and each side of the zirconia specimens was light-cured for 40 s with a LED polymerizing unit (Optilight Max; Gnatus Equipamentos Médico-Odontológicos, São Paulo, Brazil) with a light intensity of 1200 mW/cm^2. The assemblies (composite resin + zirconia + composite resin) were left undisturbed for 5 min to

complete the self-curing. The assemblies, in a number of 6 for each group, were stored in distilled water at a temperature of 37ºC before sectioning.

Thirty microbar specimens with cross-sectioned areas of 1 mm^2 were achieved for each group through two perpendicular sections using a precision cutting device (Isomet, Buehler, An ITW Company, IL, USA) and a diamond-coated saw (IsoMet Blade, 15HC, 127mm; Buehler, An ITW Company, IL, USA). Abundant water cooling was employed during the cutting, which was performed with a maximum speed of 300rpm to reduce microbars loss.

Hydrothermal aging

Half of the bonded microbar specimens were stored in distilled water at room temperature for 24 h before μTBS testing. The other half was subjected to hydrothermal aging for 10,000 cycles (521-4D, Ethik Technology, São Paulo, Brazil) between two water baths, one at 5ºC and the other at 55ºC, with a dwell time of 30 s.

Microtensile bond strength test (μTBS)

The microbar specimens were measured with a digital caliper and then attached to the equipment test fixation unit using cyanoacrylate glue (Almasuper; Almata Ind. e Com. Ltda., Curitiba, Brazil), taking care to equally locate the resin-to-zirconia interfaces in the free space of the attachment unit. The bond strength test was performed with a universal testing machine (Instron 4444; Instron®, MA, USA) at a crosshead speed of 1.0mm/min until bond failure. The data were obtained in (N) and stored in the equipment software (Bluehill 3; Instron®, MA, USA).

Scanning electron microscopy and adhesive analysis

All fractured microbars specimens were gold sputter coated and examined under SEM (HITACHI Tabletop Microscope TM3030; Hitachi, Ltd., Tokyo, Japan) to assess the failure modes. Although there were two adhesive interfaces, only the side that suffered the failure was evaluated. The failures were classified as adhesive (partial or complete failure in the adhesive or debonding of the adhesive from the surface), cohesive (partial or complete failure in the ceramic or

composite), or mixed failures (combination of adhesive/cohesive failure).[21,53] A quantitative assessment of the adhesive remnant area (ARA) of all fractured microbars was also calculated using the ImageJ open-source image analysis software (Version 1.51m9; National Institutes of Health, MD, USA).

Data analysis

To analyze the surface roughness differences between groups, the R_a values of each block were evaluated by the two-way ANOVA and Sydak's tests (α=0.05). Zirconia phase fractions were identified by the Rietveld refinement method using the MAUD (Materials Analysis Using Diffraction) software.[54]

The bond strength values (MPa) were measured by calculating the applied loads at failure (N) obtained from the testing machine and then divided by the cross-section areas (mm^2) of each microbar. Differences between the means were analyzed using one-way ANOVA and Tukey's tests. Also, the Weibull modulus (m) and characteristic strength (σ_0) were calculated from the values of bond strength (MPa), in order to represent the accumulative distribution and probability density of each group. The following equation was used:

$$pf = 1 - exp\left[-\left(\frac{\sigma}{\sigma_0}\right)m\right]$$

where $pf(\sigma)$ is the probability of failure, σ is the fracture strength, σ_0 is the characteristic strength at the fracture probability of 63.2% and m is the Weibull modulus. The Weibull parameters were calculated based on the maximum likelihood method. Afterwards, the Weibull modulus (m) and characteristic strength (σ_0) were statistically evaluated using the Weibull analysis.

The ARA values were obtained calculating the surface areas of the remnant cementing agent on each microbar after the fracture, SEM images were analyzed through ImageJ software to this purpose. Next, these areas were transformed into percentages and the mean and standard deviation for each group were calculated.

RESULTS

Mean and the standard deviation values of surface roughness (R_a) for the six groups and the results of the two-way ANOVA and Sidak's tests are listed in

Table 2. According to the analysis, there were significant statistical differences between groups, being the ZrC-AO and ZrT-AO the groups with the highest roughness. The X-ray diffractograms were similar for both conventional and high-translucent zirconia and are shown in Fig. 2. The Rietveld data are detailed in Table 3. The analysis revealed a higher content of cubic zirconia (c-ZrO_2) phase for the high-translucent zirconia (50,6 wt%), while a minimum content was identified for the conventional zirconia (2,4 wt%).

Microtensile bond streght (μTBS), characteristic strength of bond strength and Weibull modulus (*m*) for groups are presented in Table 4. The μTBS as well as the characteristic strength of the ZrC-AO group was significantly higher than those observed for all other groups. Mechanically treated conventional zirconia groups (ZrC-AO, ZrC-T) showed higher μTBS and characteristic strength when compared to the corresponding translucent zirconia groups (ZrT-AO, ZrT-T). Microbars specimens without surface conditioning showed the lowest mean values for both conventional and high-translucent zirconia groups.

In relation to the results found for the Weibull modulus (m), the highest values were found for the ZrC-AO, ZrC-T, ZrT-AO e ZrT-T groups, respectively, but without statistical differences between them, showing that, for these groups, the data distribution is narrower and therefore more reliable.[55] No significant effect of hydrothermal aging on the characteristic strength was observed the experimental groups, however, for the ZrT group, a significant drop in characteristic strength was observed after aging.

Data regarding failure mode and ARA of each group are shown in Table 5 and graphically presented by the SEM images in Figs 3 and 4 where adhesive and mixed failures are represented. The most frequent type of failure between the groups was mixed failure, however, neither group presented cohesive failure. The ZrT group had the highest percentage of adhesive failures (as sintered: 80 %; after hydrothermal aging: 92.31 %), as well as the lowest ARA (as sintered: 4.52 ± 8.45 %; after hydrothermal aging: 4.48 ±7.09 %).

DISCUSSION

The present study evaluated the bond strength of conventional and high-translucent zirconia. Based on the results obtained, the first null hypothesis tested for the present study was partially rejected. High-translucent zirconia presented

lower values of bond strength when compared to the conventional zirconia groups. Only the ZrT group was statistically similar to the ZrC group both as sintered and after hydrothermal treatment. After hydrothermal treatment only ZrC-AO and ZrT-AO groups were statistically similar. The second null hypothesis was accepted since no differences were observed between the aged groups. However, the ZrT group showed a significant reduction on the characteristic strength after aging.

A different mechanical and optical behavior of high-translucent zirconia have been reported due to the modifications in its microstructure compared to conventional zirconia,[4,5] however, no evidence regarding a distinct adhesive performance has been reported. The higher content of cubic zirconia phase of high-translucent zirconia revealed by the XRD analysis confirmed microstructural modifications between the materials tested.[56,57] Accordingly, the data of the present study suggest that these modifications could also be related to its adhesive behavior.

Since the zirconia surface does not favor durable adhesive bonding, surface conditioning methods have been proposed in order to improve the interaction between resin-based luting cements and the zirconia surface.[22,58] Among the mechanical methods proposed in the literature for adhesion to zirconia, the most employed is the air-borne particle abrasion using Al_2O_3 particles.[9,29,44] Air-borne particle abrasion is responsible for removing the contamination from surfaces, creating a rough surface that increases the surface area and improves the surface energy.[11] For air-borne abrasion, Sciasci et al. reported that the use of particles ranging from 30 to 120 μm could lead to similar results.[58] In the present study, particles of 50 μm were used to inhibit the formation defects or microcracks, which can occur when larger particles are used.[11,59] Tribochemical treatment is another method widely used to improve the adhesive bond to zirconia, creating a more reactive surface.[32] This silica-enriched surface is capable to favorable react with the adhesive systems.[32] To enable a durable bond strength, the association of MDP has also been proposed since it promotes a water resistant chemical bond to metal oxides.[27,28,31] Lately, universal adhesives presenting both silane and phosphate ester monomers in their composition have been developed.[33]

The groups without any surface treatment (ZrC and ZrT) evaluated in the

present study showed the lowest bond strength. However, between treated groups, air-particle abraded specimens had higher bond strength values than those that received tribochemical treatment with silica-coated alumina particles. These findings could be explained by the roughness values measured for the ZrC-T and ZrT-T groups that were statistically similar to those obtained for the ZrC and ZrT groups (Table 2). In addition, no silanization was performed, since the manufacturer of the universal adhesive used in this study does not recommend the use of silane separately, as it already contains silane in its composition. However, the silane contained in universal adhesives could have reduced capacity to bond to silica-enriched surfaces, due to silanol condensation.[60,61] Even when the roughness values were statistically similar, the different μTBS values between these groups suggests that tribochemical treatment has a positive effect on the adhesive behavior.

Salem et al. reported favorable results of adhesive strength of translucent zirconia when air-particle abrasion is performed combined with the use of an MDP-containing universal adhesive. The results of adhesive resistance found for the group treated with air-particle abrasion were higher (28.9 ± 4.2 MPa) than those found in the present study, however, the roughness of the specimens was also higher (6.4 ± 0.9 μm) compared to the roughness of the ZrC-AO (0.364 ± 0.032 μm) and ZrT-AO (0.353 ±0.024 μm) groups.

Hydrothermal aging was chosen since it would effectively simulate interface aging.[9,38,51] The worst case aging scenario would be represented after 10,000 cycles.[9,62] Only a slight effect on the bond strength values after hydrothermal aging was registered, without statistical significance for all groups. These results suggest a durable bond to conventional and high-translucent zirconia when micromechanical and chemical treatments are performed. These findings are in agreement with studies by Lüthy et al. and Kumbuloglu et al. where no significant differences were found for conventional zirconia after thermocycling when surface treatments and chemical adhesion through MPD was performed.[36,37] Regarding high-translucent zirconia, similar remarks were reported by Salem et al., however, the aging method employed by the authors was water storage for 3 months.[39]

The μTBS test allows a better alignment of the specimens and more homogeneous distribution of stress, which results in a more sensitive analysis of

adhesive strength, with failure occurring at the weakest link of the bonding interface.[9,38,41,44,45] In the present study, an alternative approach was proposed to overcome the inherent difficulties of cutting an extremely hard material such as zirconia. Through this experimental methodology, it was possible to reduce the amount of zirconia needed to perform the tests and facilitate the sectioning steps while maintaining the integrity of the bonded interfaces. However, due to this methodology, two interfaces are present and since the failure occurred on only one of the interfaces, it was not possible to evaluate the behavior of the second interface. Through the proposed methodology, 30 specimens for each group were achieved after sectioning the assemblies, however, until completing this objective, some pre-test failures occurred. The debonding percentages at this stage were approximately as follows: ZrC (20%); ZrC-AO (0%); ZrC-T (5%); ZrT (45%); ZrT-AO (4%) and ZrT-T (9%). It is also important to note that, after sectioning, specimens that were not submitted to aging showed failures during the μTBS test (ZrC: 3, ZrT:8, and ZrT-T: 2 specimens). The ZrC-AO, ZrC-T and ZrT-AO groups showed no failure during the test. After thermal aging, only one specimen from the ZrC group and seven from the ZrT group debonded. During the μTBS test, a single aged specimen from the ZrC and ZrT-T groups and two specimens from the ZrT group failed.

The pre-test failures found in the present study, during the cut of the specimens, after thermocycling and during the adhesive test, did not impair the analysis of the results. A study by Júnior et al.[50] showed pre-test failures of 100% in two of the zirconia groups evaluated when prepared by the traditional technique. Other studies also reported a high rate of pre-test failures when using the conventional μTBS technique,[48,49] which sometimes makes it impossible to compare the results. Thus, although it is still an experimental methodology that needs to be better studied and validated, the initial results are promising.

The most prevalent type of failure in the groups studied was mixed failure. Adhesive failures were more evident in the ZrT group without aging and in the ZrC and ZrT groups after aging. Cohesive failures were not found. However, in order to detail better the results regarding the failure mode, the mixed failures were further described by means of a modification to the adhesive remnant index (ARI) initially proposed by Årtum & Bergland[63] in 1984. In the present study, the adhesive remnant area (ARA) test was proposed, which allowed a more accurate

evaluation of the area occupied by the remnant cementing agent in the specimens through the digital treatment of the SEM images. ARA showed areas between (32.15 ± 16.60%) and (66.85 ± 8.66%) in the groups that received mechanical treatment and between (4.48 ± 7.09%) and (23.37 ± 18.36) in the untreated groups. These results agree with the values of adhesive resistance.

The data obtained regarding bond strength were homogeneous for all groups and in accordance with similar studies from the literature,[39,43,64] which could validate this experimental approach for future studies. However, it is possible to highlight the limited number of materials studied, the use of a single adhesive cement, the lack of silanization of the groups subjected to tribochemical treatment, as well as the absence of mechanical aging (i.e., chewing simulation) as limitations of the present study. More studies are needed to understand the adhesive behavior of translucent zirconia, as well as to better evaluate the interface (zirconia - composite resin) and the effect of the changes in the microstructure of the material in relation to the adhesive strength.

CONCLUSIONS

Within the limitations of the present study, it is possible to conclude that the bond strength values of the mechanically treated high-translucent zirconia groups were lower than those obtained for conventional zirconia counterparts. A durable bond strength to both materials was achieved after mechanical treatment by air-borne particle abrasion or tribochemical treatment in combination with an MDP-containing universal adhesive and an adhesive resin cement. For silica-enriched zirconia surfaces, silanization would be recommended even when using MDP-containing universal adhesives.

Clinical Significance

Mechanical surface treatment (air-borne particle abrasion or tribochemical treatment) associated with the use of universal adhesives containing MDP could provide a durable bonding to conventional and high-translucent zirconia.

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Table 1: Materials Used.

Material	Code	Composition (wt%)	Density after sintering (g/cm³)	Average grain size (μm)	Manufacturer
Conventional zirconia	HT Blank	ZrO_2 + HfO_2 + Y_2O_3 ≥99%, Y_2O_3 4.5%~6%, Al_2O_3 ≤0.5%, other oxides ≤0.5%	6.07 ± 0.01	0.4	Upcera; Shenzhen Upcera Dental Technology Co., Liaoning, China
High-translucent zirconia	TT Blank	ZrO_2 + HfO_2 86.3%~94.2%, Y_2O_3 9.7%, Er_2O_3 <2%, Fe_2O_3 <0.5%, Al_2O_3 <0.5%, other oxides <0.5%	≥6.0	0.4	Upcera; Shenzhen Upcera Dental Technology Co., Liaoning, China

Table 2: Results of the statistical analysis of surface roughness values (R_a) in micrometers (µm) (mean ± S.D.).

Material	Surface Treatment		
	No treatment	AO	T
Conventional zirconia (ZrC)	0.227 ± 0.023^{Aa}	0.364 ± 0.032^{Ab}	0.247 ± 0.026^{Aa}
High-translucent zirconia (ZrT)	0.255 ± 0.084^{Aa}	0.353 ± 0.024^{Ab}	0.283 ± 0.027^{Aa}

* Superscript uppercase letters indicate a significant difference between materials, and superscript lowercase letters indicate a significant difference between surface treatments ($p<0.05$)

Table 3. Zirconia phase fractions (wt%)

Material	t-ZrO2	c-ZrO2	m-ZrO2
Conventional zirconia (ZrC)	96.1	2.5	1.4
High-translucent zirconia (ZrT)	47.4	50.6	2.0

Table 4 – Microtensile bond strength (μTBS) (mean ± S.D.), characteristic strength (σ_0) and Weibull modulus (m) of conventional and high-translucent zirconia groups. (95% confidence interval)

	Bond Strength (MPa)				Characteristic Strength (σ_0) (MPa)		Weibull Modulus (m)	
	As Sintered	n	Hydrothermal Aging	n	As Sintered	Hydrothermal Aging	As Sintered	Hydrothermal Aging
ZrC	8.71 (± 5.47) $_{d}$	27	6.64 (± 3.83) $_{de}$	28	11.53 (9.76-13.63) $_{g,i}$	8.34 (7.08-9.82) $_{i,j,k}$	2.58 (1.87-3.57) $_{d,e,f,g,h,i}$	2.47 (1.81-3.37) $_{d,e,f,g,h,i,j}$
ZrC-AO	20.86 (± 5.12) $_{a}$	30	19.99 (± 5.87) $_{ab}$	30	22.78 (21.10-24.59) $_{a}$	22.01 (20.05-24.16) $_{a,b}$	4.91 (3.68-6.55) $_{a,b,c}$	4.01 (3.00-5.36) $_{a,b,c,d,e,f}$
ZrC-T	16.71 (± 5.18) $_{b}$	30	18.77 (± 4.09) $_{ab}$	30	18.45 (16.78-20.29) $_{b,c,e}$	20.31 (19.06-21.64) $_{a,b,c}$	3.92 (2.89-5.31) $_{a,b,c,d,e,f,g}$	5.90 (4.40-7.91) $_{a,b}$
ZrT	7.14 (± 3.76) $_{de}$	22	3.38 (± 2.10) $_{e}$	21	8.98 (7.62-10.58) $_{h,i,j}$	4.58 (3.85-5.45) $_{l}$	2.03 (1.50-2.75) $_{h,i,j,k}$	1.27 (0.92-1.75) $_{k,l}$
ZrT-AO	16.39 (± 4.36) $_{b}$	30	15.18 (± 4.04) $_{bc}$	30	18.24 (17.14-19.41) $_{c,d,e}$	16.70 (15.29-18.23) $_{d,e,f}$	6.17 (4.59-8.29) $_{a}$	4.29 (3.25-5.68) $_{a,b,c,d,e}$
ZrT-T	9.81 (± 4.30) $_{d}$	28	11.35 (± 3.88) $_{cd}$	29	11.70 (10.48-13.07) $_{f,g,h,i}$	12.92 (11.85-14.08) $_{g}$	3.52 (2.60-4.76) $_{a,b,c,d,e,f,g,h}$	4.42 (3.26-5.99) $_{a,b,c,d}$

* Values followed by the same letters are statistically similar ($p>0.05$).

Table 5 - Adhesive remnant area (ARA) and recorded failure mode.

	% of mixed failure*		ARA (%)	
	As Sintered	Hydrothermal Aging	As Sintered	Hydrothermal Aging
ZrC	66.67	18.52	23.37 (±18.36)	7.41 (±10.88)
ZrC-AO	100.00	100.00	66.85 (±8.66)	62.28 (±14.10)
ZrC-T	92.31	100.00	53.00 (±17.71)	58.00 (±6.61)
ZrT	20.00	7.69	4.52 (±8.45)	4.48 (±7.09)
ZrT-AO	100.00	100.00	58.22 (±11.85)	54.18 (±9.82)
ZrT-T	90.00	95.45	32.15 (±16.60)	41.13 (±14.62)

*The rest percentage represents adhesive failures.

Figure Legends:

Fig. 1 - Schematic diagram showing the followed methodology.

Fig 2. - XRD patterns of conventional (ZrC) and high-translucent zirconia (ZrT).

Fig. 3 - Representative SEM images of all groups without hydrothermal aging at magnifications of 120x and 500x. Adhesive failure mode of the ZrT group while mixed failure mode for all other groups can be observed.

Fig. 4 - Representative SEM images of all groups subjected to hydrothermal aging (t) at magnifications of 120x and 500x. Adhesive failure mode of the ZrC and ZrT groups while mixed failure mode of the ZrC-AO, ZrC-T, ZrT-AO and ZrT-T groups can be observed.

Table 1 – Mean and standard deviation (coefficient of variation) of tensile strength σ_0 and Weibull Modulus (95% confidence interval) (m).

	Tensile Strength (σ_0) (Mpa)		Weibull Modulus (m)	
	As Sintered	Hydrothermal Aging	As Sintered	Hydrothermal Aging
ZrC	11.53 (9.76-13.63) $_{g,i}$	8.34 (7.08-9.82) $_{i,j,k}$	2.58 (1.87-3.57) $_{d,e,f,g,h,i}$	2.47 (1.81-3.37) $_{d,e,f,g,h,i,j}$
ZrC-AO	22.78 (21.10-24.59) $_{a}$	22.01 (20.05-24.16) $_{a,b}$	4.91 (3.68-6.55) $_{a,b,c}$	4.01 (3.00-5.36) $_{a,b,c,d,e,f}$
ZrC-T	18.45 (16.78-20.29) $_{b,c,e}$	20.31 (19.06-21.64) $_{a,b,c}$	3.92 (2.89-5.31) $_{a,b,c,d,e,f,g}$	5.90 (4.40-7.91) $_{a,b}$
ZrT	8.98 (7.62-10.58) $_{h,i,j}$	4.58 (3.85-5.45) $_{l}$	2.03 (1.50-2.75) $_{h,i,j,k}$	1.27 (0.92-1.75) $_{k,l}$
ZrT-AO	18.24 (17.14-19.41) $_{c,d,e}$	16.70 (15.29-18.23) $_{d,e,f}$	6.17 (4.59-8.29) $_{a}$	4.29 (3.25-5.68) $_{a,b,c,d,e}$
ZrT-T	11.70 (10.48-13.07) $_{f,g,h,i}$	12.92 (11.85-14.08) $_{g}$	3.52 (2.60-4.76) $_{a,b,c,d,e,f,g,h}$	4.42 (3.26-5.99) $_{a,b,c,d}$

* Same letters mean statistically similar behavior (α=0.05).

Table 2: Adhesive remnant area (ARA) and recorded failure mode.

	% of mixed failure*		ARA	
	As Sintered	Hydrothermal Aging	As Sintered	Hydrothermal Aging
ZrC	66.67	18.52	23.37 (±18.36)	7.41 (±10.88)
ZrC-AO	100.00	100.00	66.85 (±8.66)	62.28 (±14.10)
ZrC-T	92.31	100.00	53.00 (±17.71)	58.00 (±6.61)
ZrT	20.00	7.69	4.52 (±8.45)	4.48 (±7.09)
ZrT-AO	100.00	100.00	58.22 (±11.85)	54.18 (±9.82)
ZrT-T	90.00	95.45	32.15 (±16.60)	41.13 (±14.62)

*The rest percentage represents adhesive failures.

Table 3: Results of the statistical analysis of surface roughness values (R_a) in micrometers (μm). The same letters in the same column indicates no significant differences according to Tukey's test ($p>0.05$)

Groups	n	Mean	Standard deviation
ZrC	6	0.23 a	±0.023
ZrC-AO	6	0.36 b	±0.032
ZrC-T	6	0.25 a	±0.026
ZrT	6	0.26 a	±0.084
ZrT-AO	6	0.35 b	±0.023
ZrT-T	6	0.28 a	±0.026

Fig. 1 - Schematic diagram showing the followed methodology

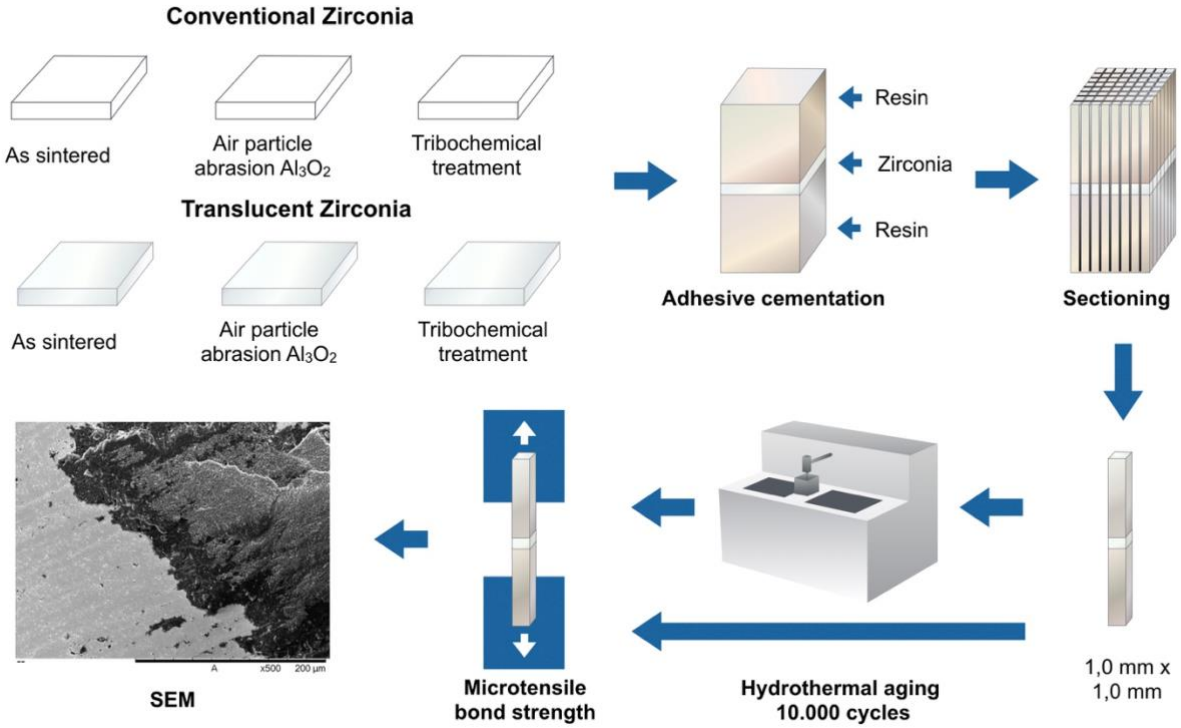


Fig 2. XRD patterns of samples before bonding procedures: A) without surface treatment (ZrC and ZrT), B) aluminum oxide air-particle abrasion with 50um particles (ZrC-OA and ZrT-OA), and C) Tribochemical treatment with silica-coated alumina particles of 50um (ZrC-T and ZrT-T).

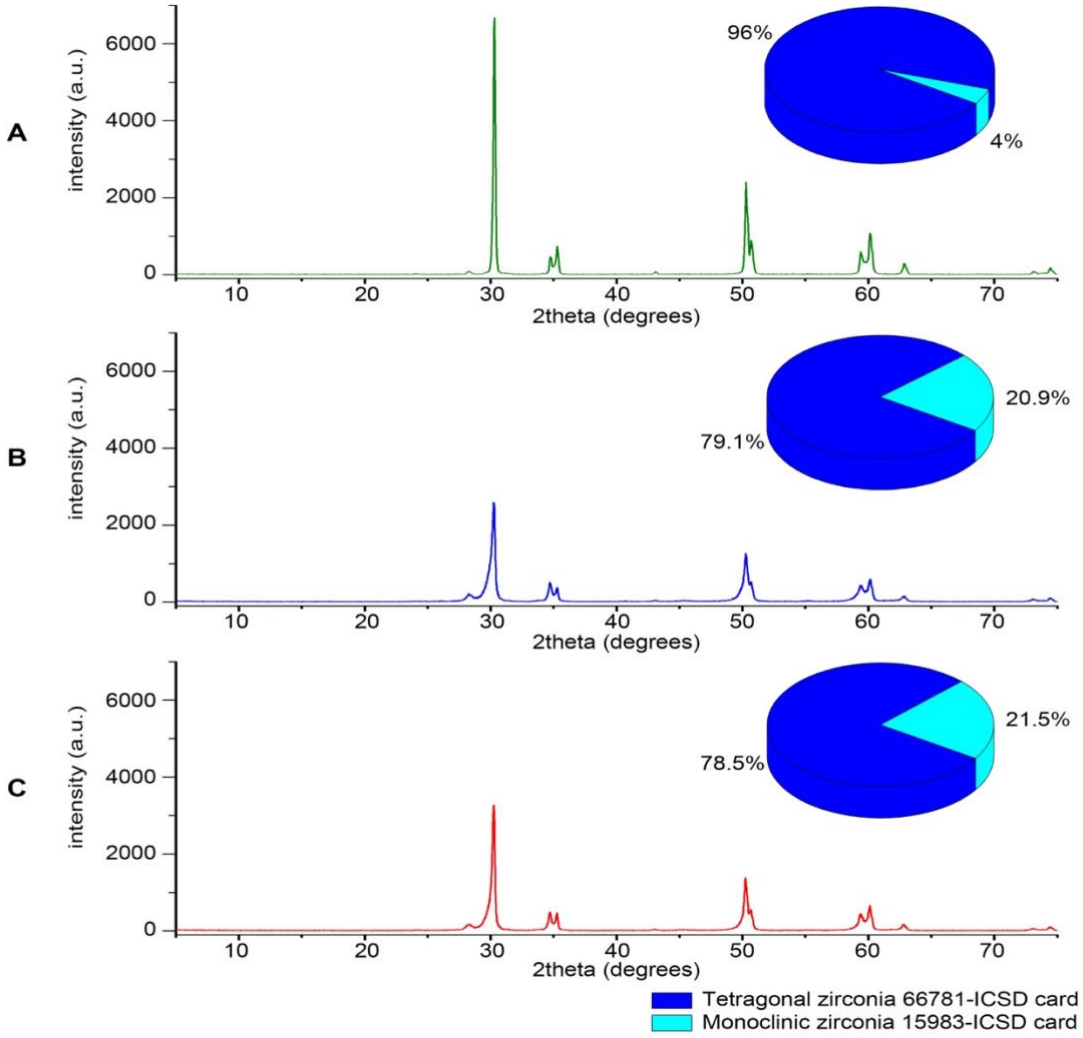
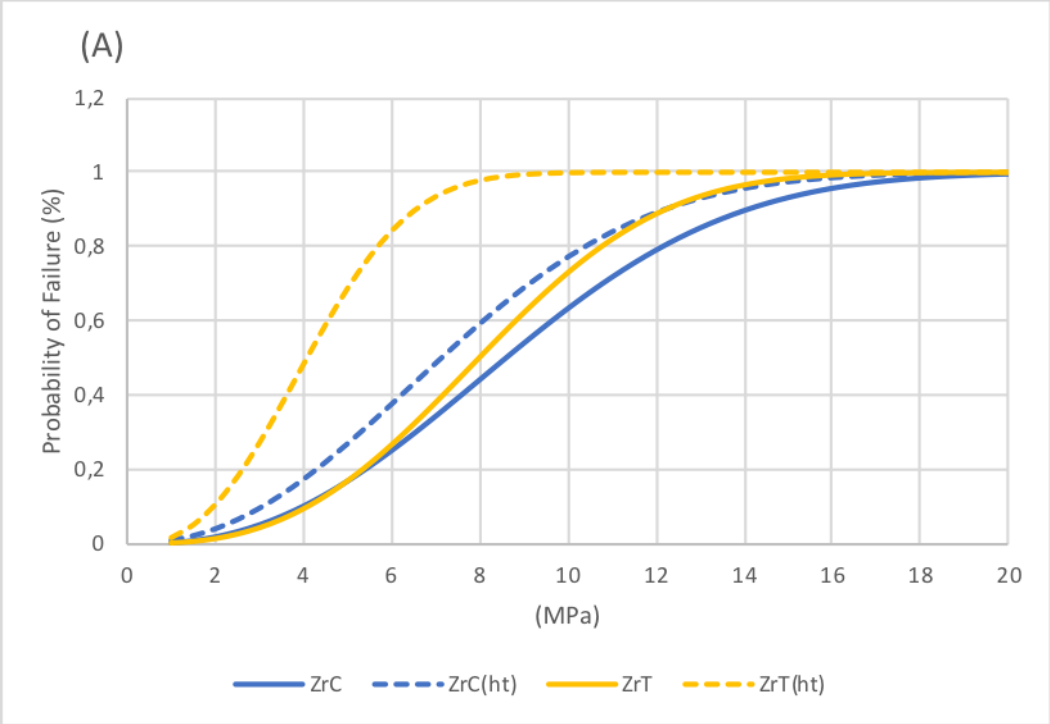


Fig. 3 - Weibull analysis representing the accumulative distribution (A) and probability density (B) of ZrC and ZrT groups before and after hydrothermal aging.



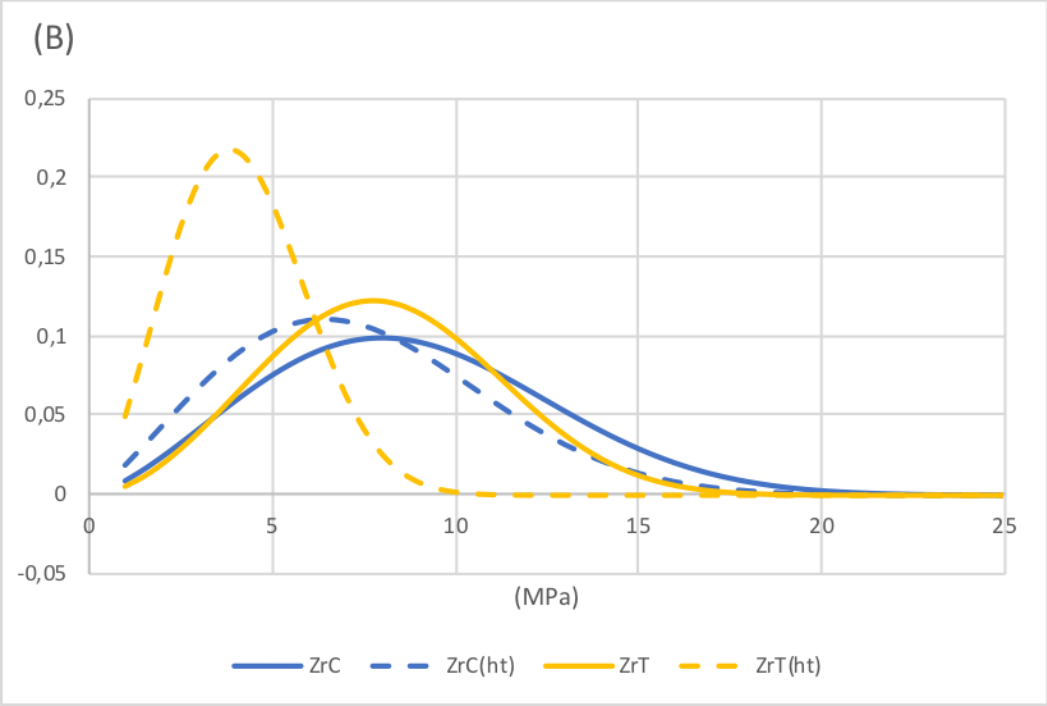


Fig. 4 - Weibull analysis representing the accumulative distribution (A) and probability density (B) of ZrC-AO and ZrT-AO groups before and after hydrothermal aging.

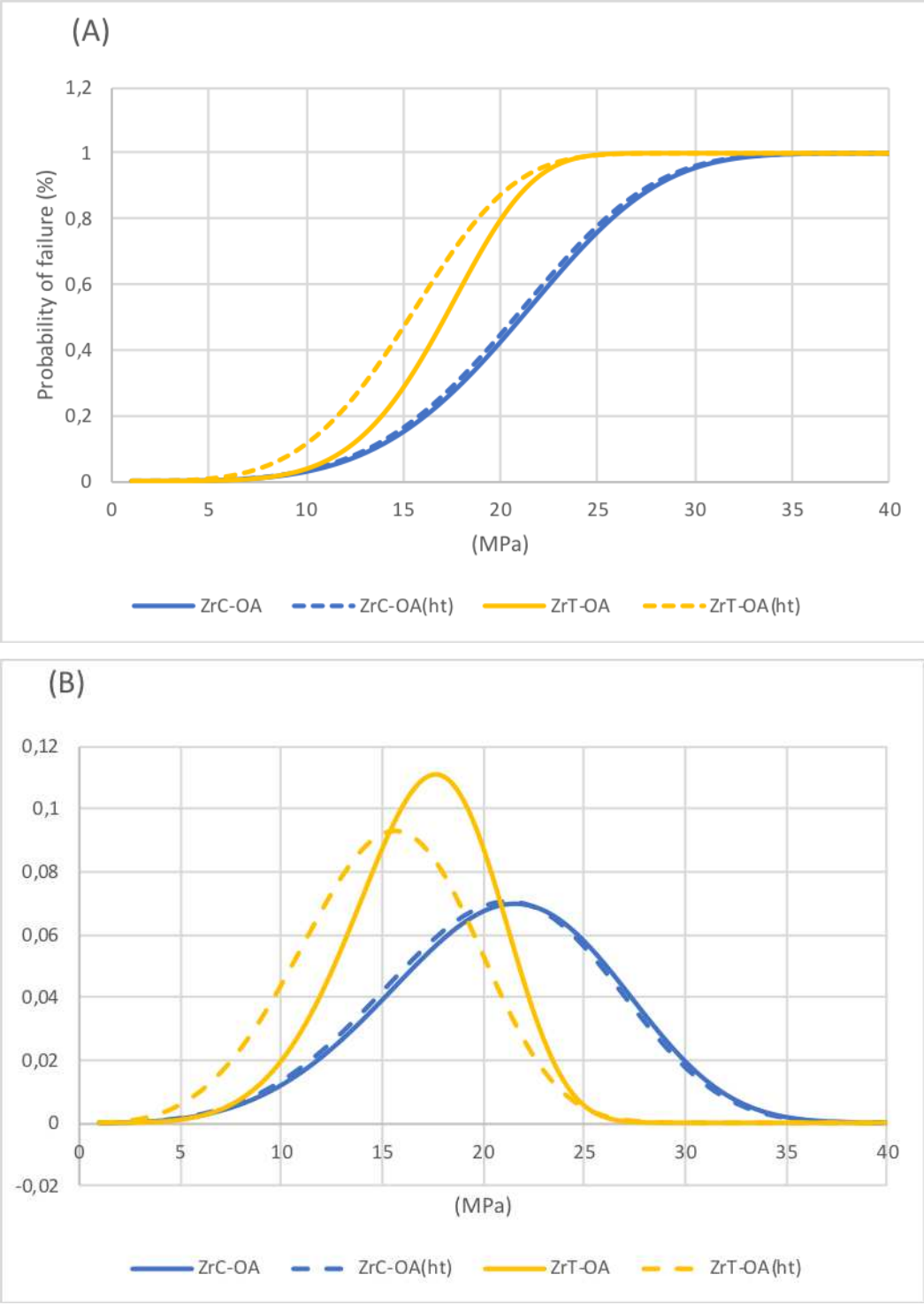


Fig. 5 - Weibull analysis representing the accumulative distribution (A) and probability density (B) of groups ZrC-T and ZrT-T groups before and after hydrothermal aging.

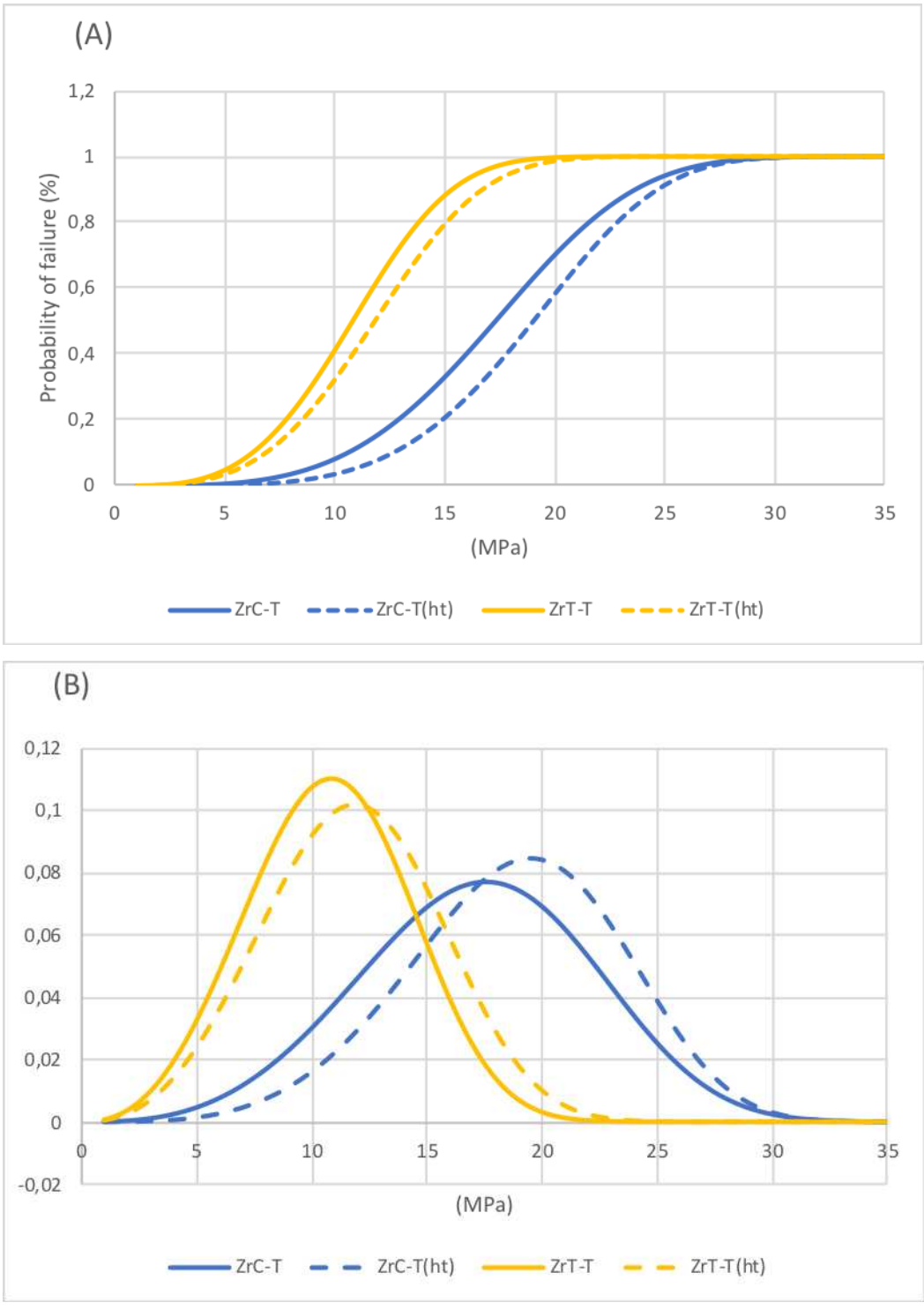


Fig. 6 - Representative SEM images of all groups without hydrothermal aging at magnifications of x120 and x500. Adhesive failure mode of ZrT group while mixed failure mode on all other groups can be seen.

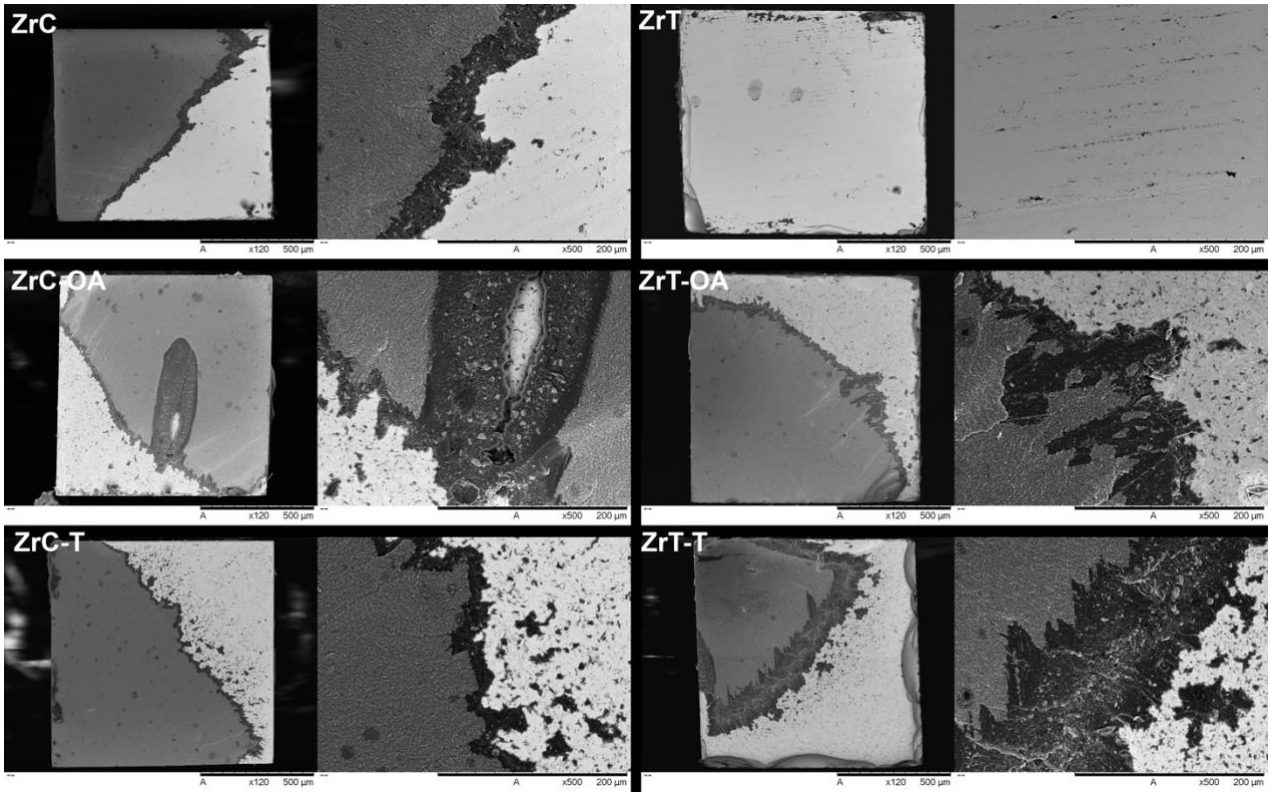


Fig. 7 - Representative SEM images of all groups subjected to hydrothermal aging (t) at magnifications of x120 and x500. Adhesive failure mode of ZrC and ZrT groups while mixed failure mode of ZrC-AO, ZrC-T, ZrT-AO and ZrT-T groups can be seen.